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# **Integrated Data Collection Analysis (IDCA) Program —KClO<sub>3</sub> (as received)/Icing Sugar**

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## **ABSTRACT**

The Integrated Data Collection Analysis (IDCA) program is conducting a proficiency study for Small-Scale Safety and Thermal (SSST) testing of homemade explosives (HMEs). Described here are the results for impact, friction, electrostatic discharge, and differential scanning calorimetry analysis of a mixture of KClO<sub>3</sub> as received from the manufacturer mixed with icing sugar, sized through a 100-mesh sieve—KClO<sub>3</sub>/icing sugar (AR) mixture. This material was selected because of the challenge of performing SSST testing of a mixture of two solid materials. The mixture was found to: 1) be more sensitive to impact than RDX, similar to PETN, 2) be the same or less sensitive to friction than PETN, and 3) to be less sensitive to spark than RDX. The thermal analysis showed that the mixture has thermally stability similar to RDX and is perhaps more energetic upon decomposition but variable results indicate sampling issues. Compared to the 100-mesh sieved counter part, the KClO<sub>3</sub>/icing sugar (-100) mixture, the AR mixture was found to be about the same sensitivity towards impact, friction and ESD.

This effort, funded by the Department of Homeland Security (DHS), ultimately will put the issues of safe handling of these materials in perspective with standard military explosives. The study is adding SSST testing results for a broad suite of different HMEs to the literature. Ultimately the study has the potential to suggest new guidelines and methods and possibly establish the SSST testing accuracies needed to develop safe handling practices for HMEs. Each participating testing laboratory uses identical test materials and preparation methods wherever possible. Note, however, the test procedures differ among the laboratories. The results are compared among the laboratories and then compared to historical data from various sources. The testing performers involved for the KClO<sub>3</sub>/icing sugar (as received) mixture are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Naval Surface Warfare Center, Indian Head Division (NSWC IHD), and Air Force Research Laboratory (AFRL/RXQL). These tests are conducted as a proficiency study in order to establish some consistency in test protocols, procedures, and experiments and to understand how to compare results when these things cannot be made consistent.

**Keywords:** Small-scale safety testing, proficiency test, round-robin test, safety testing protocols, HME, RDX, potassium chlorate, sugar.



# 1 INTRODUCTION

The IDCA Proficiency Test was designed to assist the explosives community in comparing and perhaps standardizing inter-laboratory Small-Scale Safety and Thermal (SSST) testing for improvised explosive materials (homemade explosives or HMEs) and aligning these procedures with comparable testing for typical military explosives<sup>1</sup>. The materials for the Proficiency Test have been selected because their properties invoke challenging experimental issues when dealing with HMEs. Many of these challenges are not normally encountered with military type explosives. To a large extent, the issues are center on the physical forms and stability of the improvised materials.

Often, HMEs are formed by mixing oxidizer and fuel precursor materials, and typically, the mixture components are combined shortly before use. The challenges to produce a standardized inter-laboratory sample are primarily associated with mixing and sampling. For solid-solid mixtures, the challenges primarily revolve around adequately mixing two powders on a small scale, producing a mixture of uniform composition—particle size and dryness often being a factor—as well as taking a representative sample. For liquid-liquid mixtures, the challenges revolve around miscibility of the oxidizer with the fuel causing the possibility of multiphase liquid systems. For liquid-solid mixtures, the challenges revolve around the ability of the solid phase to mix completely with the liquid phase, as well as minimizing the formation of intractable or ill-defined slurry-type products.

The IDCA has chosen several formulations to test that present these challenges. Table 1 shows the materials selected for the Proficiency Test and the Description column describes the form of the resulting mixture.

**Table 1. Materials for IDCA Proficiency study**

Oxidizer/Explosive	Fuel	Description
Potassium perchlorate	Aluminum	Powder mixture
Potassium perchlorate	Charcoal	Powder mixture
Potassium perchlorate	Dodecane <sup>1</sup>	Wet powder
Potassium chlorate	Dodecane <sup>1</sup>	Wet powder
Potassium chlorate as received	Sucrose (icing sugar mixture) <sup>2,3</sup>	Powder mixture
Potassium chlorate -100 mesh <sup>3</sup>	Sucrose (icing sugar mixture) <sup>2,3</sup>	Powder mixture
Sodium chlorate	Sucrose (icing sugar mixture) <sup>2,3</sup>	Powder mixture
Ammonium nitrate		Powder
Bullseye <sup>®</sup> smokeless powder <sup>4</sup>		Powder
Ammonium nitrate	Bullseye <sup>®</sup> smokeless powder <sup>4</sup>	Powder mixture
Urea nitrate	Aluminum	Powder mixture
Urea nitrate	Aluminum, sulfur	Powder mixture
Hydrogen peroxide 70%	Cumin	Viscous paste
Hydrogen peroxide 90%	Nitromethane	Miscible liquid
Hydrogen peroxide 70%	Flour (chapatti)	Sticky paste
Hydrogen peroxide 70%	Glycerine	Miscible liquid
HMX Grade B		Powder
RDX Class 5 Type II		Powder (standard)
PETN Class 4		Powder (standard)

1. Simulates diesel fuel; 2. Contains 3 wt % cornstarch; 3. Sieved to pass 100 mesh; 4. Alliant Bullseye<sup>®</sup> smokeless pistol gunpowder;

Evaluation of the results of SSST testing of unknown materials, such as the HMEs in Table 1, is generally done as a relative process, where a well understood standard is tested alongside the HME. In many cases, the standard employed is PETN or RDX. The standard is obtained in a high purity, narrow particle size range, and measured frequently. The performance of the standard is well documented on the same equipment (at the

testing laboratory), and is used as the benchmark. The sensitivity to external stimuli and reactivity of the HME (or any energetic material) are then evaluated relative to the standard.

Most of the results from SSST testing of HMEs are not analyzed any further than this. The results are then considered in-house. This approach has worked very well for military explosives and has been a validated method for developing safe handling practices. However, there has never been a validation of this method for HMEs. Although it is generally recognized that these SSST practices are acceptable for HME testing, it must always be kept in mind that HMEs have different compositional qualities and reactivities than conventional military explosives.

The IDCA is attempting to evaluate SSST testing methods as applied to HMEs. In addition, the IDCA is attempting to understand, at least in part, the laboratory-to-laboratory variation that is expected when examining the HMEs. The IDCA team has taken several steps to make this inter-laboratory data comparison easier to analyze. Each participating laboratory uses materials from the same batches and follows the same procedures for synthesis, formulation, and preparation. In addition, although the Proficiency test allows for laboratory-to-laboratory testing differences, efforts have been made to align the SSST testing equipment configurations and procedures to be as similar as possible, without significantly compromising the standard conditions under which each laboratory routinely conducts their testing.

The first and basic step in the Proficiency test is to have representative data on a standard material to allow for basic performance comparisons. Table 1 includes some standard military materials. Class 5 Type II RDX was chosen as the primary standard, and Class 4 PETN was chosen as a secondary material. These materials are being tested in triplicate and RDX will continue to be tested throughout the IDCA Proficiency test.

The subject of this report,  $\text{KClO}_3$ /icing sugar (AR) mixture, is the third in a series of materials that fall in the class of solid oxidizer/fuel mixtures. These materials were chosen for study in the Proficiency test because of the challenge of testing fine solids mixed with fuels—adequate mixing on a small scale and representative sampling of a physical mixture. In contrast to the previous study on  $\text{KClO}_3$ /icing sugar, where both precursors were sized through a 100-mesh sieve<sup>2</sup>, the mixture in this present study was prepared from precursors that only the icing sugar was sized through a 100-mesh sieve, while the  $\text{KClO}_3$  was used as received from the manufacturer. This was done to note the effect of particle size on test results.

The testing performers in this work are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), and Naval Surface Warfare Center, Indian Head Division (NSWC IHD) and the Air Force Research Laboratory (AFRL/RXQL).

## 2 EXPERIMENTAL

*General information.* All samples were prepared according to the IDCA Program report on drying and mixing procedures<sup>3,4</sup>. The  $\text{KClO}_3$  was obtained from Columbus Chemical as a purified powder, Catalog #4230PV, Lot # 200917615, CAS # 3811-04-9, assay (COA by manufacturer):  $\text{KClO}_3$ , 99.7%;  $\text{KCl}$ , 0.05%;  $\text{H}_2\text{O}$ , 0.05%. The icing sugar was obtained from a local food market as C & H Icing (powdered) sugar, Lot # 79174-A14. No manufacturer analysis was given on the container, but the C & H sugar web site<sup>5</sup> lists the icing sugar as having 3% cornstarch added as an anti-caking agent. DHS SNL provided elemental composition from combustion analysis and Karl Fischer assay: C,  $41.70 \pm 0.05\%$ ; H,  $6.24 \pm 0.10\%$ ; N,  $0.35 \pm 0.25\%$ ; O,  $51.49 \pm 0.48\%$ ; moisture,  $0.29 \pm 0.01\%$ ; residual  $0.21 \pm 0.29\%$ <sup>6</sup>. Both precursors were dried for 16 h and cooled in a desiccator according to IDCA drying methods<sup>4</sup>. The sugar was sieved through a minus 100-mesh (150  $\mu\text{m}$  hole size) sieve and the  $\text{KClO}_3$  was used as received from the manufacturer. The mixture was prepared by hand, mixing the two solids together in a materials compatible polypropylene container according to

IDCA mixing and compatibility procedures<sup>3</sup>. The mixture composition is 74-wt % KClO<sub>3</sub> and 26-wt % icing sugar. Typically, the precursors are mixed at that ratio to give approximately a 1-gram sample. This sample is divided up for the various SSST testing. Three samples were prepared this way and tested separately. The mixing ratio was determined by thermochemical calculations using Cheetah to find the optimum detonation energy output<sup>1</sup>. The ratio chosen matched stoichiometric for oxygen balance. The aged samples were prepared by mixing the components to give a 1-g sample, and then the mixture was left undisturbed at ambient conditions until used for the measurements.

The SSST testing data for the individual participants was obtained from the following reports: LLNL IDCA Project Report—KC/Sugar (screened) [revised 4.20.11] (LLNL)<sup>7</sup>, Potassium Chlorate and Sugar 51088B, revised 4.6.11 (LANL)<sup>8</sup>, KC/Sugar Report (as received) [revised 3.30.11] (IHD)<sup>9</sup>, and Potassium Chlorate (KC) + Sugar, As Received, Integrated Data Collection Analysis (IDCA) Program, Small Scale Safety Testing (SSST) (AFRL)<sup>10</sup>.

**Table 2. Summary of conditions for the analysis of KClO<sub>3</sub>/icing sugar (AR) mixture (All = LANL, LLNL, IHD, AFRL)**

Impact Testing		8. Data analysis—LLNL, IHD, and AFRL modified Bruceton (log-scale spacing) and TIL; LANL, modified Bruceton (linear spacing) and TIL
1. Sample size—LLNL, IHD, and AFRL 35±2 mg, LANL 40±2 mg	ESD	1. Sample size—All, ~5 mg, but not weighed
2. Preparation of samples—All, dried per IDCA procedures <sup>4</sup>		2. Preparation of samples—All, dried per IDCA procedures <sup>4</sup>
3. Sample form—All, loose powder		3. Sample form—All, powder
4. Powder sample configuration—All, conical pile		4. Tape cover—LANL, scotch tape; LLNL, Mylar; IHD and AFRL, no tape
5. Apparatus—LANL, LLNL, IHD, Type 12; AFRL MBOM with type 12 tooling*		5. Sample configuration—All, cover the bottom of sample holder
6. Sandpaper—LANL, 150 grit or 180 garnet; IHD, 180 garnet; LLNL, 120 flint S/C paper; AFRL, 180 garnet		6. Apparatus—LANL, IHD, AFRL, ABL; LLNL, custom built*
7. Sandpaper size—All, 1 inch square except LANL 1.25 inch diameter disk		7. Positive detection—All, by observation
8. Drop hammer weight—All, 2.5 kg		8. Analysis methods—All, TIL
9. Positive detection—LANL and LLNL, microphones with electronic interpretation as well as observation; IHD and AFRL, observation		
10. Data analysis—All, modified Bruceton and TIL before and at threshold; LANL also uses Neyer		
Friction analysis		Differential Scanning Calorimetry
1. Sample size—All, ~5 mg, but not weighed		1. Sample size—All, ~ <1 mg
2. Preparation of samples—All, dried per IDCA procedures <sup>4</sup>		2. Preparation of samples—All, according to IDCA procedures <sup>4</sup>
3. Sample form—All, powder		3. Sample holder—All, hermetic with pin hole; LLNL, also sealed pan
4. Sample configuration—All, small circle form		4. Scan rate—All, 10°C/min
5. Apparatus—LANL, LLNL, IHD, BAM; IHD, AFRL, ABL*		5. Range—All, 40 to 400°C
6. Positive detection—All, by observation		6. Pan hole size—LLNL 50 µm; LANL, IHD, and AFRL, 75 µm
7. Room Lights—LANL and AFRL on; LLNL off; IHD (BAM) on, (ABL) off		7. Instruments—LANL, TA Instruments Q2000; LLNL, TA Instruments 2920; IHD, TA Instruments Q1000, AFRL TA Instruments Q2000*

Footnotes: \*Test apparatus, *Impact*: LANL, LLNL, IHD—ERL Type 12 Drop Weight Sensitivity Apparatus, AFRL—MBOM modified for ERL Type 12 Drop Weight; *Friction*: LANL, LLNL, IHD—BAM Friction Apparatus, LANL, IHD, AFRL—ABL Friction Apparatus; *Spark*: LANL, IHD, AFRL—ABL Electrostatic Discharge Apparatus, LLNL—custom-built Electrostatic Discharge Apparatus; *Differential Scanning Calorimetry*: LANL—TA Instruments Q1000, Q2000, LLNL—TA Instruments 2910, 2920, Setaram Sensys DSC, IHD—TA Instruments Model 910, 2910, Q1000, AFRL—TA Instruments Q2000.

*Testing conditions.* Table 2 summarizes the SSST testing conditions used by the laboratories that participated in the analyses of the  $\text{KClO}_3$ /icing sugar (AR) mixture.

### 3 RESULTS

#### 3.1 $\text{KClO}_3$ /icing sugar (AR) mixture

In this proficiency test, all testing participants are required to use materials from the same batch, and mixtures are to be prepared by the same methods. However, the actual testing procedures can be different. These differences are described in the IDCA report on method comparisons<sup>11</sup>, which compares the different procedures by each testing category. LANL, LLNL, IHD, and AFRL participated in this part of the SSST testing of the  $\text{KClO}_3$ . The  $\text{KClO}_3$  was used as received from the manufacturer so the effect of particle size of the oxidizer could be compared. A previous report was issued<sup>2</sup> where the oxidizer was screened to pass through a 100-mesh sieve. Although  $\text{KClO}_3$  and sugar mixtures can be found at a variety of mixing ratios, the ratio for this study was selected that conforms to the maximum energy output, as determined by thermochemical assessments.

#### 3.2 Impact testing results for $\text{KClO}_3$ /icing sugar (AR) mixture

Table 3 shows the results of impact testing of the  $\text{KClO}_3$ /icing sugar (AR) mixture as performed by LANL, LLNL, IHD, and AFRL. Differences in the testing procedures are shown in Table 2, and the notable differences are the sandpaper grit size, amount of sample, and the methods for detection of a positive event. All participants performed data analysis by normal modified Bruceton method<sup>12,13</sup> and LANL also performed data analysis by the Neyer method<sup>14</sup>.

**Table 3. Impact testing results for  $\text{KClO}_3$ /icing sugar (AR) mixture**

Lab <sup>1</sup>	Test Date	T, °C	RH, % <sup>2</sup>	DH <sub>50</sub> , cm <sup>3</sup>	s, cm <sup>4</sup>	s, log unit <sup>4</sup>
LLNL (120)	3/11/10	22.7	18	14.9	0.858	0.025
LLNL (120)	3/12/10	23.3	24	15.0	1.002	0.029
LLNL (120)	3/15/10	23.3	18	17.0	3.229	0.082
LANL (150)	3/1/10	22.1	15.1	15.3	4.685	0.131
LANL (150)	3/2/10	22.3	16.0	13.4	1.515	0.049
LANL (150)	3/3/10	21.8	12.2	16.3	1.843	0.049
LANL (180)	4/28/10	22.5	< 10	11.0	3.581	0.139
LANL (180)	4/29/10	21.5	< 10	10.7	2.612	0.105
LANL (180)	5/4/10	21.0	< 10	9.5	0.942	0.043
IHD (180)	8/4/10	20	45	9	2.519	0.120
IHD (180)	8/12/10	20	50	11	2.296	0.090
IHD (180)	8/19/10	20	50	11	1.781	0.070
AFRL (180)	5/7/10	26.7	54	10.2	1.4	0.060
AFRL (180)	6/23/10	26.1	57	6.9	1.9	0.123
AFRL (180)	6/23/10	25.6	57	7.9	2.8	0.161

1. Number in parentheses indicates grit size of sandpaper; 2. Relative humidity; 3. Modified Bruceton method, load, in cm, for 50% reaction (DH<sub>50</sub>); 4. Standard deviation.

The test results from the four participating laboratories for impact show a range for DH<sub>50</sub> from 6.9 to 17.0 cm. The average values are, in cm: LLNL,  $15.6 \pm 1.2$ ; LANL,  $12.7 \pm 2.7$ ; IHD,  $10.3 \pm 1.2$ ; AFRL,  $8.3 \pm 1.7$ . The average values based on sandpaper grit size are, in cm: 120,  $15.6 \pm 1.2$ ; 150,  $15.0 \pm 1.5$ ; 180,  $9.7 \pm 1.5$ . The average values for 180-grit sandpaper are, in cm: LANL,  $10.4 \pm 0.8$ ; IHD,  $10.3 \pm 1.2$ ; AFRL,  $8.3 \pm 1.7$ . The standard deviation is below the 0.1 log unit range except for IHD and AFRL, where the value is over 0.1 log



units. This appears as a result of IHD using 0.1 log spaced steps while LANL and LLNL use 0.05 log spaced steps. AFRL uses linear spaced steps. The impact of step spacing will be evaluated in detail in a later report.

Table 4 shows the impact test results from LANL using the Neyer or D-Optimal method<sup>14</sup>. The  $DH_{50}$  values are in the same range as the values analyzed by the Bruceton method, where the averages for the Neyer method are, in cm:  $14.9 \pm 0.7$  cm and  $10.5 \pm 1.4$  cm for the tests that used 150-grit and 180-grit sandpaper, respectively.

**Table 4. Impact testing results for  $KClO_3$ /icing sugar (AR) mixture (Neyer or D-Optimal Method)**

Lab <sup>1</sup>	Test Date	T, °C	RH, % <sup>2</sup>	$DH_{50}$ , cm <sup>3</sup>	s, cm <sup>4</sup>	s, log unit <sup>4</sup>
LANL (150)	3/1/10	21.7	16.3	15.3	0.9	0.026
LANL (150)	3/2/10	22.2	16.5	14.1	1.5	0.046
LANL (150)	3/3/10	21.0	16.0	15.3	0.6	0.017
LANL (180)	4/28/10	22.3	< 10	11.5	1.73	0.065
LANL (180)	4/29/10	21.5	11	11.1	2.34	0.091
LANL (180)	5/4/11	22.2	< 10	8.9	1.63	0.079

1. Number in parentheses indicates grit size of sandpaper; 2 Relative humidity; 3. Neyer method, load, in cm, for 50% reaction ( $DH_{50}$ ); 4. Standard deviation.

### 3.3 Friction testing results for $KClO_3$ /icing sugar (AR) mixture

**Table 5. BAM Friction Testing results for  $KClO_3$ /icing sugar (AR) mixture**

Lab	Test Date	T, °C	RH, % <sup>1</sup>	TIL, kg <sup>2</sup>	TIL, kg <sup>3</sup>	$F_{50}$ , kg <sup>4</sup>	s, kg <sup>5</sup>	s, log unit <sup>5</sup>
LLNL	3/11/10	22.2	18	0/10 @ 11.2	1/10 @ 12.0	11.5	1.32	0.050
LLNL	3/12/10	22.2	18	0/10 @ 8.0	1/10 @ 8.4	14.3	3.53	0.106
LLNL	3/15/10	22.2	18	0/10 @ 7.2	1/10 @ 8.0	9.7	1.84	0.082
LLNL <sup>6</sup>	10/6/10	22.8	20	0/10 @ 6.0	1/10 @ 7.2	14.3	0.76	0.023
LANL	3/1/10	21.8	13.8	NA <sup>7</sup>	NA <sup>7</sup>	< 4.7	NA <sup>8</sup>	NA <sup>8</sup>
LANL	3/2/10	22.7	15.3	NA <sup>7</sup>	NA <sup>7</sup>	4.9	1.97	0.17
LANL	3/3/10	22.2	12.0	NA <sup>7</sup>	NA <sup>7</sup>	< 4.3	NA <sup>8</sup>	NA <sup>8</sup>
LANL	3/1/10	22.1	15.1	Too low	2/10 @ 2.4	NA <sup>9</sup>	NA <sup>9</sup>	NA <sup>9</sup>
LANL	3/2/10	22.7	15.3	Too low	3/10 @ 2.4	NA <sup>9</sup>	NA <sup>9</sup>	NA <sup>9</sup>
LANL	3/3/10	22.2	12.0	Too low	2/10 @ 2.4	NA <sup>9</sup>	NA <sup>9</sup>	NA <sup>9</sup>
IHD	7/21/10	29	39	0/10 @ 2.5	1/1 @ 2.9	NA <sup>9</sup>	NA <sup>9</sup>	NA <sup>9</sup>
IHD	7/17/10	29	40	0/10 @ 2.9	1/1 @ 3.3	NA <sup>9</sup>	NA <sup>9</sup>	NA <sup>9</sup>
IHD	7/20/10	29	38	0/10 @ 2.9	1/2 @ 3.3	NA <sup>9</sup>	NA <sup>9</sup>	NA <sup>9</sup>
IHD	8/20/10	26	46	NA <sup>7</sup>	NA <sup>7</sup>	3.5	1.06	0.130
IHD	8/20/10	26	46	NA <sup>7</sup>	NA <sup>7</sup>	3.5	0.68	0.084
IHD	8/20/10	26	46	NA <sup>7</sup>	NA <sup>7</sup>	3.9	0.73	0.081

1. Relative humidity; 2. Threshold Initiation Level (TIL) is the load (kg) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 3. Next level where positive initiation is detected; 4. Modified Bruceton method, load, in kg, for 50% probability of reaction ( $F_{50}$ ), LLNL and IHD use log spacing; LANL uses linear spacing; 5. Standard Deviation; 6. LLNL used LANL plates; 7. Not applicable, separate sample used for TIL analysis; 8. The material shows a positive at 2.4 kg, the lowest value available on the LANL BAM friction tester with the 2.4 kg spacing scale (linear spacing). The statistical evaluation is not valid as a result; 9. Not applicable, separate sample used for Bruceton analysis.

Table 5 shows the BAM Friction testing performed by LANL, LLNL and IHD. AFRL does not have BAM friction testing. The difference in testing procedures by the three laboratories is shown in Table 2, and the notable differences are in the methods for positive detection. All participants performed data analysis using the threshold initiation level method (TIL)<sup>15</sup>, and a modified Bruceton method<sup>12</sup>. The average friction values for  $F_{50}$  are, in kg: LLNL,  $11.8 \pm 2.3$ ; LANL, 4.9; IHD,  $3.6 \pm 0.2$ . The standard deviation values range for all



0.02 < log s < 0.13. The threshold values are in the following order IHD  $\approx$  LANL < LLNL. For TIL, 0 positive events, LANL could not measure that low; while LLNL have higher values than IHD. LLNL also tested the KClO<sub>3</sub>/icing sugar (AR) mixture in the LLNL apparatus, but using plates obtained from LANL testing. The measured values show little difference from the values derived using LLNL plates—perhaps the threshold values are on the lower side.

Table 6 shows the ABL Friction testing performed by IHD and AFRL on the KClO<sub>3</sub>/icing sugar (AR) mixture. IHD and AFRL were the only participants to report ABL Friction testing results. LANL did not have the system in routine performance at the time. LLNL does not have ABL Friction. The results show the F<sub>50</sub> is about 150  $\pm$  11 psig at 8 fps (IHD values). The threshold values vary significantly depending on the participant.

**Table 6. ABL Friction testing results for KClO<sub>3</sub>/icing sugar (AR) mixture**

Lab	Test Date	T, °C	RH, % <sup>1</sup>	TIL, psig/fps <sup>2,3</sup>	TIL, psig/fps <sup>2,4</sup>	F <sub>50</sub> , psig/fps <sup>2,5</sup>	s, psig/fps <sup>6</sup>	s, log unit <sup>6</sup>
IHD	8/10/10	27	45	0/20 @ 100/8	1/6 @ 135/8	NA <sup>7</sup>	NA <sup>7</sup>	NA <sup>7</sup>
IHD	8/10/10	27	42	0/20 @ 75/8	1/3 @ 100/8	NA <sup>7</sup>	NA <sup>7</sup>	NA <sup>7</sup>
IHD	8/10/10	27	43	0/20 @ 100/8	1/4 @ 135/8	NA <sup>7</sup>	NA <sup>7</sup>	NA <sup>7</sup>
IHD	8/11/10	27	42	NA <sup>8</sup>	NA <sup>8</sup>	139/8	63/8	0.19
IHD	8/11/10	26	44	NA <sup>8</sup>	NA <sup>8</sup>	160/8	52/8	0.14
IHD	8/11/10	26	43	NA <sup>8</sup>	NA <sup>8</sup>	152/8	69/8	0.19
AFRL	5/6/10	26.7	58	0/10 @ 42/6	1/4 @ 56/6	NA <sup>7</sup>	NA <sup>7</sup>	NA <sup>7</sup>
AFRL	5/7/10	26.7	55	0/10 @ 42/6	4/7 @ 56/6	NA <sup>7</sup>	NA <sup>7</sup>	NA <sup>7</sup>
AFRL	6/25/10	26.1	55	0/10 @ 13/6	1/5 @ 18/6	NA <sup>7</sup>	NA <sup>7</sup>	NA <sup>7</sup>

1. Relative humidity; 2. psig/fps = pressure in psig at test velocity in feet per sec; 3. Threshold Initiation Level (TIL) is the load, in psig at # fps, at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 4. Next level where positive initiation is detected; 5. Modified Bruceton method, load, in psig at # fps, for 50% Reaction (F<sub>50</sub>); 6. Standard deviation; 7. Not applicable, TIL only was performed; 8. Not applicable, F<sub>50</sub> measurements only were performed.

### 3.4 Electrostatic discharge testing of KClO<sub>3</sub>/icing sugar (AR) mixture

**Table 7. Electrostatic discharge testing KClO<sub>3</sub>/icing sugar (AR) mixture**

Lab	Test Date	T, °C	RH, % <sup>1</sup>	TIL, Joule <sup>2</sup>	TIL, Joule <sup>3</sup>
LLNL <sup>4</sup>	3/11/10	22.2	18	0/10 @ 1.0	0/10 @ 1.0
LLNL <sup>4</sup>	3/12/10	22.8	18	0/10 @ 1.0	0/10 @ 1.0
LLNL <sup>4</sup>	3/15/10	22.8	18	0/10 @ 1.0	0/10 @ 1.0
LANL	3/1/10	21.8	13.8	0/20 @ 0.125	2/2 @ 0.250
LANL	3/2/10	22.3	16.4	0/20 @ 0.125	2/4 @ 0.250
LANL	3/3/10	21.8	12.2	0/20 @ 0.125	2/4 @ 0.250
IHD	7/17/10	29	40	0/20 @ 0.326	1/1 @ 0.853
IHD	7/20/10	29	38	0/20 @ 0.326	1/1 @ 0.853
IHD	7/20/10	29	38	0/20 @ 0.165	1/3 @ 0.326
AFRL	5/6/10	22.2	43	0/20 @ 0.088	1/1 @ 0.13
AFRL	5/7/10	26.8	55	0/20 @ 0.099	1/1 @ 0.13
AFRL	5/7/10	26.8	45	0/20 @ 0.088	1/1 @ 0.13

1. Relative humidity; 2. Threshold Initiation Level (TIL) is the load (joules) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 3. Next level where positive initiation is detected; 4. LLNL uses a 510-ohm resistor in the discharge unit to mimic the human body.

Electrostatic Discharge (ESD) testing of the KClO<sub>3</sub>/icing sugar (AR) mixture was performed by LANL, IHD, AFRL and LLNL. Table 7 shows the results. Differences in the testing procedures are shown in Table 2, and

the notable differences are the use of tape and what covers the sample. In addition, LLNL uses a custom built ESD system with a 500- $\Omega$  series resistor in line to simulate a human body, making a direct comparison of the data from LLNL with data generated by the other participants challenging. (LLNL has purchased a new ABL spark tester and is being used for the spark testing on the 3<sup>rd</sup> RDX calibration run and the remaining IDCA threats.) . All participants performed data analysis using the threshold initiation level method (TIL)<sup>15</sup>.

Each laboratory finds the KClO<sub>3</sub>/icing sugar (AR) mixture to have a different sensitivity to ESD. The order is AFRL > IHD > LANL > LLNL. The data from LLNL indicate a non-sensitive material. This is expected because of the LLNL experimental configuration.

### 3.5 Thermal testing (DSC) of KClO<sub>3</sub>/icing sugar (AR) mixture

Differential Scanning Calorimetry (DSC) was performed on the KClO<sub>3</sub>/icing sugar (AR) mixture by LLNL, LANL, AFRL, and IHD. All participating laboratories used different versions of the DSC by TA Instruments. Table 8 shows the DSC exothermic and endothermic values. For all four participants there is observed a sharp, low-temperature exothermic feature, Ex<sub>1</sub>, with T<sub>max</sub> values ranging from 173.5 to 183.6 °C. The data from AFRL exhibit a second exothermic feature, Ex<sub>2</sub>, with a T<sub>max</sub> around 219°C. LANL and LLNL data (not shown) also exhibit this feature in some DSC profiles of this material. All participants observed, at least in some samples, a third exothermic feature, Ex<sub>3</sub>, with a T<sub>max</sub> between 334.9 to 336.5°C. In some cases, this exothermic feature was so sharp that the instrument control program could not determine the onset temperature.

**Table 8. Differential Scanning Calorimetry results for KClO<sub>3</sub>/icing sugar (AR) mixture (pinhole hermetic pan), 10°C/min heating rate**

Lab	Test Date	Exothermic (Ex <sub>1</sub> ), onset/maximum, °C ( $\Delta H$ , J/g)	Exothermic (Ex <sub>2</sub> ), onset/maximum, °C ( $\Delta H$ , J/g)	Exothermic (Ex <sub>3</sub> ), onset/maximum, °C ( $\Delta H$ , J/g)
LLNL	3/10/10	175.8/182.9 (3021)	NA <sup>1</sup>	NA <sup>1</sup>
LLNL	3/10/10	178.3/183.4 (2294)	NA <sup>1</sup>	NA <sup>1</sup>
LLNL	3/11/10	177.2/183.6 (2841)	NA <sup>1</sup>	NA <sup>1</sup>
LANL	3/1/10	174.5/176.0 (957)	NA <sup>1</sup>	334.7/336.2 (1167)
LANL	3/1/10	None <sup>2</sup> /180.6 (2774)	NA <sup>1</sup>	NA <sup>1</sup>
LANL	3/2/10	177.1/178.1 (929)	NA <sup>1</sup>	332.3/335.6 (446)
LANL	3/2/10	176.8/177.8 (591)	NA <sup>1</sup>	334.2/334.9 (1172)
LANL	3/4/10	None <sup>2</sup> /180.0 (2585)	NA <sup>1</sup>	NA <sup>1</sup>
LANL	3/4/10	None <sup>2</sup> /179.3 (1492)	NA <sup>1</sup>	332.8/336.2 (184)
IHD	3/18/11	171.9/173.5 (3210)	NA <sup>1</sup>	NA <sup>1</sup>
IHD	3/18/11	173.7/175.2 (1578)	NA <sup>1</sup>	NA <sup>1</sup>
IHD	3/18/11	173.4/174.7 (1908)	NA <sup>1</sup>	NA <sup>1</sup>
AFRL	6/25/11	176.2/177.4 (597)	198.8/219.5 (369)	None/335.9 (1005)
AFRL	6/25/11	176.5/178.1 (601)	196.2/218.0 (474)	334.5/336.5 (957)
AFRL	6/28/11	None <sup>2</sup> /179.8 (1454)	NA <sup>1</sup>	NA <sup>1</sup>

1. Not observed in this set of data; 2. Not discerned by instrument control program.

The overall  $\Delta H$  values (when adding contributions from Ex<sub>1</sub> + Ex<sub>2</sub> + Ex<sub>3</sub>) are all well over 1000 J/g for the samples. However, the distribution of the enthalpy among the exothermic features is sample dependent. When there is only one feature, which is always Ex<sub>1</sub>, the enthalpy is obviously not divided. However, when there are other features, the  $\Delta H$  varies from sample to sample, with no discernable trend.

All participants observed instrument limitations that manifest as a positive slope on the cooling part of the Ex<sub>1</sub> feature. This feature is due to an equipment limitation that is discussed below, but probably impacts the accu-

racy of the enthalpy measurement. The full extent of these issues with the DSC of this material will be discussed in detail elsewhere.

Table 9 shows the DSC data, by LLNL only, for the  $\text{KClO}_3$ /icing sugar (AR) mixture where the sample holder is closed instead of pinhole vented used for the measurements shown in Table 8. The exothermic features are very similar to comparable data from the pinhole sample holder, shown in Table 8, with the  $\text{Ex}_1$ ,  $\text{Ex}_2$ , and  $\text{Ex}_3$  having similar  $T_{\text{max}}$  and  $\Delta H$  values, when present. The total enthalpy values (sum of  $\text{Ex}_1$ ,  $\text{Ex}_2$ , and  $\text{Ex}_3$ ) are also well over 1000 J/g, similar to Table 8. The data also shows some interesting temporal qualities, where the 0 h sample exhibits only  $\text{Ex}_1$ , the 124 h sample exhibits both  $\text{Ex}_1$  and  $\text{Ex}_3$ , while the most aged sample exhibit  $\text{Ex}_1$ ,  $\text{Ex}_2$ , and  $\text{Ex}_3$ . Note, that  $\text{Ex}_2$  has also been seen by LANL and LLNL as well as AFRL, in un-aged samples. Whether the temporal history of the sample is important has yet to be determined, but the origin of this behavior will be discussed in detail in a future report.

**Table 9. Differential Scanning Calorimetry results for  $\text{KClO}_3$ /icing sugar (AR) mixture (closed hermetic pan),  $10^\circ\text{C}/\text{min}$  heating rate**

Lab	Aging Time, h	Exothermic ( $\text{Ex}_1$ ), onset/maximum, $^\circ\text{C}$ ( $\Delta H$ , J/g)	Exothermic ( $\text{Ex}_2$ ), onset/maximum, $^\circ\text{C}$ ( $\Delta H$ , J/g)	Exothermic ( $\text{Ex}_3$ ), onset/maximum, $^\circ\text{C}$ ( $\Delta H$ , J/g)
LLNL	0	176.5/183.0 (3450)	NA <sup>1</sup>	NA <sup>1</sup>
LLNL	124	177.6/179.1 (1161)	NA <sup>1</sup>	332.9/337.8 (509)
LLNL	148	175.7/177.4 (591)	201.0/222.0 (943)	340.8/343.9 (284)
LLNL	152	176.7/178.5 (653)	199.8/221.7 (1139)	340.3/348.7 (543)

1. Not observed in this set of data.

## 4 DISCUSSION

Table 10 shows the average values for the data from each participant and compares it to corresponding data for standards, RDX and PETN. The data for RDX comes from the IDCA first iterative study of RDX as part of this Proficiency Test<sup>16</sup>. The data for PETN was provided by the participating laboratories (when available) from measurements performed outside this Proficiency Test. The data for the  $\text{KClO}_3$ /icing sugar (-100) mixture comes from the previous material studied in the Proficiency Test using the same components<sup>2</sup>. The only difference in the materials is that  $\text{KClO}_3$  in that study was screened through a 100-mesh sieve in that previous study.

### 4.1 Sensitivity of $\text{KClO}_3$ /icing sugar (AR) mixture compared to standards

*Impact sensitivity.* Although the impact sensitivity varies among the participating laboratories for the  $\text{KClO}_3$ /icing sugar (AR) mixture, the overall trend is that it is more impact sensitive than RDX, roughly similar to PETN.

*Friction sensitivity.* Although LLNL results for BAM friction do not agree with LANL and IHD results, when compared to the RDX standard, the  $F_{50}$  friction values for  $\text{KClO}_3$ /icing sugar (AR) mixture are significantly lower indicating it is more sensitive to friction. The only threshold data for PETN comes from LLNL and comparison with the  $\text{KClO}_3$ /icing sugar (AR) mixture threshold data would suggest that it is less sensitive, than PETN.

For ABL friction measurements, IHD and AFRL are the participants that provided any data that can be compared to standards. When examining the data from IHD only, the  $\text{KClO}_3$ /icing sugar (AR) mixture appears to be about as sensitive as RDX, inconsistent with the BAM friction results. The data from AFRL, exhibits the opposite compared to RDX, where the  $\text{KClO}_3$ /icing sugar (AR) mixture is much more sensitive, in agreement with the BAM friction results. Currently, there is no ABL friction data for PETN.

**Table 10. Average Comparison values**

	LLNL	LANL	IHD	AFRL
Impact Testing <sup>1</sup>	DH <sub>50</sub> , cm	DH <sub>50</sub> , cm	DH <sub>50</sub> , cm	DH <sub>50</sub> , cm
KClO <sub>3</sub> /icing sugar (AR) <sup>2</sup>	15.6 <sup>3,4</sup>	12.7 <sup>5,6</sup>	10.3 <sup>3,7</sup>	8.3 <sup>3,7</sup>
KClO <sub>3</sub> /icing sugar (-100) <sup>8</sup>	14.8 <sup>4</sup>	14.0 <sup>6</sup>	14.3 <sup>7</sup>	ND <sup>9</sup>
RDX Class 5 Type II <sup>10</sup>	24.1 <sup>4</sup>	25.4 <sup>11</sup>	19 <sup>7</sup>	15.3 <sup>7</sup>
PETN <sup>12</sup>	15	14.7	ND <sup>9</sup>	ND <sup>9</sup>
BAM Friction Testing <sup>13,14</sup>	TIL, kg; F <sub>50</sub> , kg	TIL, kg; F <sub>50</sub> , kg	TIL, kg; F <sub>50</sub> , kg	TIL, kg; F <sub>50</sub> , kg
KClO <sub>3</sub> /icing sugar (AR) <sup>15</sup>	9.5 <sup>16</sup> ; 11.8 <sup>16</sup>	2.4 <sup>16</sup> ; 4.9 <sup>17</sup>	3.2 <sup>16</sup> ; 3.6 <sup>16</sup>	ND <sup>9</sup> ; ND <sup>9</sup>
KClO <sub>3</sub> /icing sugar (-100) <sup>8</sup>	6.9; 9.9	4.8; 5.8	2.3; 4.4	ND <sup>9</sup> ; ND <sup>9</sup>
RDX Class 5 Type II <sup>10</sup>	19.2; 25.1	21.6; 20.8	16.8; ND <sup>9</sup>	ND <sup>9</sup> ; ND <sup>9</sup>
PETN <sup>12</sup>	6.4; 10.5	ND <sup>9</sup> ; 9.2	ND <sup>9</sup> ; ND <sup>9</sup>	ND <sup>9</sup> ; ND <sup>9</sup>
ABL Friction Testing <sup>18-21</sup>	TIL, psig; F <sub>50</sub> , psig	TIL, psig; F <sub>50</sub> , psig	TIL, psig; F <sub>50</sub> , psig	TIL, psig; F <sub>50</sub> , psig
KClO <sub>3</sub> /icing sugar (AR) <sup>22</sup>	ND <sup>9</sup> ; ND <sup>9</sup>	ND <sup>9</sup> ; ND <sup>9</sup>	123 <sup>23</sup> ; 150 <sup>23</sup>	43 <sup>23</sup> ; ND <sup>9</sup>
KClO <sub>3</sub> /icing sugar (-100) <sup>8</sup>	ND <sup>9</sup> ; ND <sup>9</sup>	ND <sup>9</sup> ; ND <sup>9</sup>	30; 42	ND <sup>9</sup>
RDX Class 5 Type II <sup>10</sup>	ND <sup>9</sup> ; ND <sup>9</sup>	ND <sup>9</sup> ; ND <sup>9</sup>	74; 154	93; ND <sup>9</sup>
PETN <sup>12</sup>	ND <sup>9</sup> ; ND <sup>9</sup>	ND <sup>9</sup> ; ND <sup>9</sup>	ND <sup>9</sup>	ND <sup>9</sup>
Electrostatic Discharge <sup>24</sup>	TIL, Joules	TIL, Joules	TIL, Joules	TIL, Joules
KClO <sub>3</sub> /icing sugar (AR) <sup>25</sup>	0/10 @ 1.0 <sup>26,27</sup>	2/4 @ 0.250 <sup>27</sup>	1/2 @ 0.677 <sup>27</sup>	1/1 @ 0.677 <sup>27</sup>
KClO <sub>3</sub> /icing sugar (-100) <sup>8</sup>	0/10 @ 1.0 <sup>26</sup>	2/7 @ 0.125	1/2 @ 0.677	ND <sup>9</sup>
RDX Class 5 Type II <sup>10</sup>	1/10 @ 1.0 <sup>26</sup>	2/11 @ 0.0625	1/14 @ 0.165	1/6 @ 0.054
PETN <sup>12</sup>	1/10 @ 1.0 <sup>26</sup>	2/2 @ 0.125	ND <sup>9</sup>	ND <sup>9</sup>

1. DH<sub>50</sub>, in cm, is by a modified Bruceton method, load for 50% probability of reaction; 2. Temperature and humidity values varied during the sets of measurements (T<sub>range</sub>, °C; RH<sub>range</sub>, %) —LLNL (22.7–23.3; 18–24), LANL (21.8–22.3; 12.2–16.0), IHD (20; 45–50), AFRL (25.6–26.7, 54–57); 3. Average of three measurements from Table 3; 4. 120-grit silicon carbide sandpaper; 5. Average of six values from Table 3; 6. Three with 150-grit garnet sandpaper and three with 180-grit garnet sandpaper; 7. 180-grit garnet sandpaper; 8. From reference 2; 9. ND = Not determined; 10. From reference 16; 11. 150-grit garnet sandpaper; 12. From data taken outside of the Proficiency Test; 13. Threshold Initiation Level (TIL) is the load (kg) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 14. F<sub>50</sub>, in kg, is by a modified Bruceton method, load for 50% Reaction; 15. Temperature and humidity values varied during the sets of measurements (T<sub>range</sub>, °C; RH<sub>range</sub>, %) —LLNL (22.2; 18), LANL (21.8–22.7; 12.0–15.3), IHD (26–29; 38–46); 16. Average of three measurements from Table 5; 17. One value only from Table 5; 18. LLNL and LANL did not perform measurements; 19. Threshold Initiation Level (TIL) is the load (psig) at test velocity (fps) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 20. F<sub>50</sub>, in psig/fps, is by a modified Bruceton method, load for 50% Reaction; 21. Measurements performed at 8 fps; 22. Temperature and humidity values varied during the sets of measurements (T<sub>range</sub>, °C; RH<sub>range</sub>, %) —IHD (26–27; 42–45), AFRL (26.1–26.7; 55–58); 23. Average of three measurements from Table 6; 24. Threshold Initiation Level (TIL) is the load (joules) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 25. Temperature and humidity values varied during the sets of measurements (T<sub>range</sub>, °C; RH<sub>range</sub>, %) —LLNL (22.2–22.8; 18), LANL (21.8–22.3; 12.2–16.4), IHD (29; 38–40), AFRL (22.2–26.8, 43–55); 26. LLNL has 510-Ω series resistor in circuit; 27. Average of three measurements from Table 7.

*Spark sensitivity.* Comparing the KClO<sub>3</sub>/icing sugar (AR) mixture spark sensitivity values to the corresponding RDX values, the mixture is less spark sensitive than RDX. There are limited values on PETN, but the comparison shows the mixture to be about the same sensitivity as PETN.

*Thermal sensitivity.* The KClO<sub>3</sub>/icing sugar (AR) mixture shows one to three exothermic features depending upon the sample—Ex<sub>1</sub>, T<sub>max</sub> around 177°C; Ex<sub>2</sub>, T<sub>max</sub> 218°C; Ex<sub>3</sub>, T<sub>max</sub> around 334°C. The occurrence of the features depends upon factors such as sampling. When comparing this result to RDX (exothermic feature around 250°C with 2200 ΔH in J/g), the KClO<sub>3</sub>/icing sugar (AR) mixture has an on-set of thermal reactivity slightly below RDX, and is evaluated to be about as sensitive. In additional samples, when only one exothermic feature is observed (this occurs near 177°C), the enthalpy is similar to or more than that observed for

RDX decomposition. The behavior of the  $\text{KClO}_3$ /icing sugar (AR) mixture in DSC will be discussed in more detail elsewhere—particularly the impact of sample preparation as well as mixing. However, for SSST evaluation of this material, the low-temperature exothermic feature is the most important feature by which to evaluate the material, because it indicates a low-temperature thermal sensitivity, and is observed in all samples.

## 4.2 Comparison of results based on participants

There are differences in methodologies and equipment configurations among the participating laboratories. So comparison of results for the same test is useful to highlight any differences in SSST testing methods. Using the average values shown in Table 10, although not statistically precise, at least allows for a qualitative comparison of any trends that may be seen among the participants.

For impact testing, the data from LLNL and LANL exhibited about the same sensitivity for the  $\text{KClO}_3$ /icing sugar (AR) mixture. IHD and AFRL exhibited lower values (more sensitive). It is not clear what is the cause, but the sensitivity appears to track the humidity during the test. It is probably the sandpaper because the testing by LANL using the 180-grit garnet paper yields values very similar to those of IHD and AFRL using the same paper. When using 150-grit sandpaper, the testing by LANL yields values that are closer to those of the testing results of LLNL. Regardless, to address the sandpaper issue fully, the IDCA participants have agreed to use 180-grit sandpaper all from the same batch for future measurements. The average results for impact testing of RDX exhibited a participant-to-participant variation as well.

For BAM Friction, LLNL average values for both TIL and  $F_{50}$  indicate a less sensitive material than the comparable values from LANL and IHD. The RDX average values for  $F_{50}$ , also show that LLNL finds the material less friction sensitive than the other participants. For ABL Friction, IHD and AFRL are widely apart in the assessment, where IHD finds the  $\text{KC}$ /icing sugar (AR) mixture much less sensitive than AFRL. The reasons for this are not clear as both participants derived similar values for RDX. Further testing will be done to resolve this issue.

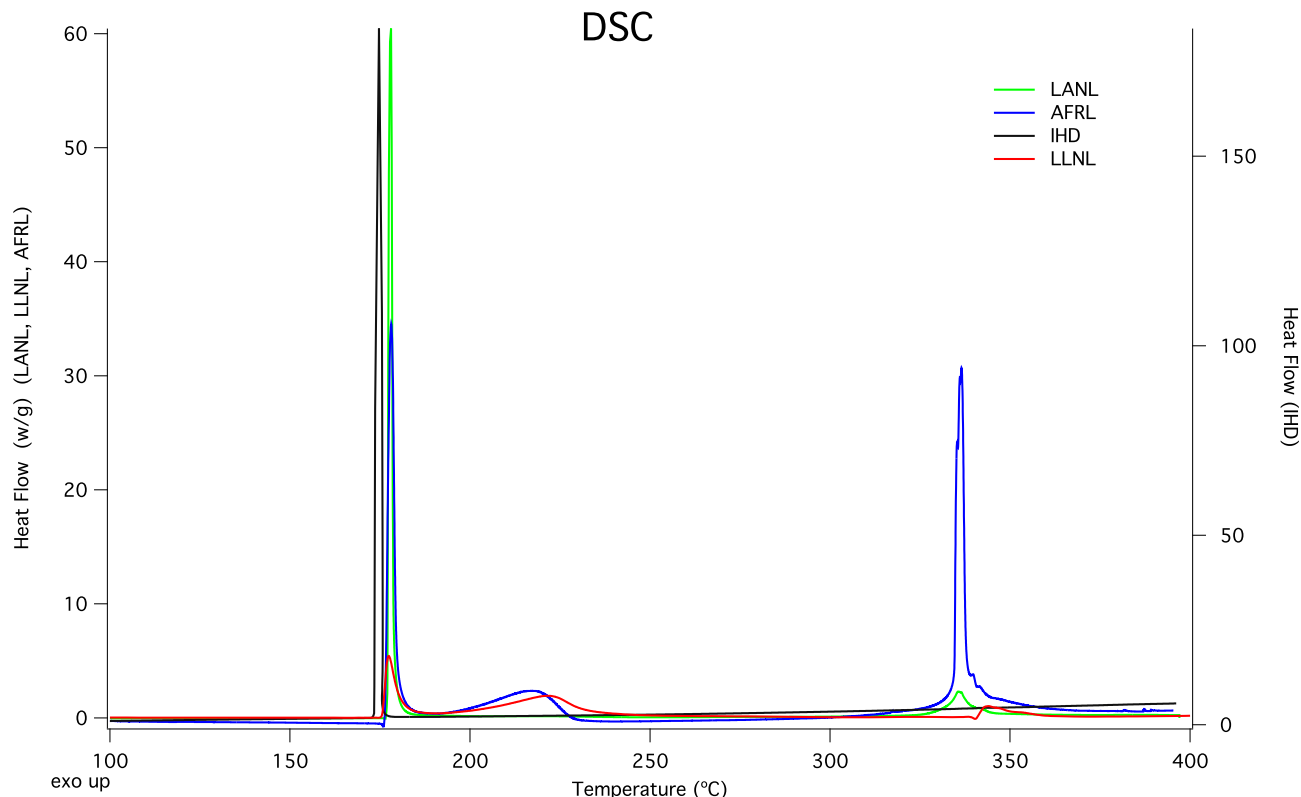
For ESD, LLNL consistently shows a much more stable material, highlighting the large design difference between the LLNL spark testing system and the others. Values from IHD and AFRL agree on  $\text{KClO}_3$ /icing sugar (AR) mixture being more stable than the values from LANL.

## 4.3 Comparisons of DSC among participants

As noted in the Results section, the DSC behavior of the  $\text{KClO}_3$ /icing sugar (AR) mixtures is highly sample dependent, where one to three exothermic features are observed. Figure 1 shows examples of the DSC profiles obtained by the different participants. Specific profiles were selected to highlight the sample variability of this material, not participant performance, and to show all features observed. The IHD profile (black line) exhibits just a single exothermic feature,  $\text{Ex}_1$ .  $\text{Ex}_1$  is observed by all the participants and is also observed in every DSC profile of the  $\text{KClO}_3$ /icing sugar (AR) mixture. This low-temperature exothermic feature seen in Figure 1 is due to a combination of the sugar melting (endothermic features) and rapid decomposition of the mixture (exothermic features) where  $\text{C}_{12}\text{H}_{22}\text{O}_{11} + 8 \text{KClO}_3 \rightarrow 8 \text{KCl} + 12 \text{CO}_2 + 11 \text{H}_2\text{O}$ . This has been documented in previous DTA and TGA experiments on  $\text{KClO}_3$  and  $\text{KClO}_4$  + sugar mixtures<sup>17,18</sup>.

The LANL profile (green) exhibits two exothermic features,  $\text{Ex}_1$  and  $\text{Ex}_3$ .  $\text{Ex}_3$  has also been seen previously. It appears to have sample-size dependence and possibly can be attributed to the reaction of the potassium chlorate with residual carbon from an incomplete oxidation that occurred during  $\text{Ex}_1$ .  $\text{KClO}_3$  alone starts to melt near  $300^\circ\text{C}$ <sup>17</sup> but does not decompose until over  $400^\circ\text{C}$ . All four participants have documented the combination of  $\text{Ex}_1$  and  $\text{Ex}_3$  in at least some samples.

The AFRL profile (blue) exhibits three exothermic features, Ex<sub>1</sub>, Ex<sub>2</sub> and Ex<sub>3</sub>. The extra feature Ex<sub>2</sub>, occurs near 220°C and is not completely understood. LANL and LLNL have also observed this exothermic feature in freshly prepared KClO<sub>3</sub>/icing sugar (AR) mixture samples and have attributed it to mixing/homogeneity issues.



**Figure 1. DSC profiles of KClO<sub>3</sub>/icing sugar (AR) mixture at 10°C/min heating rate**

The LLNL profile (red) also exhibits three exothermic features—the same as AFRL. However, the DSC profile is of a mixture that was aged for 6.3 days. Because the sample was examined in a hermetically sealed DSC sample holder, the exothermic features are slightly shifted to higher temperatures. In addition, the same material, when examined at  $t = 0$  exhibits only Ex<sub>1</sub>.

As shown in the figure, the Ex<sub>1</sub> can have an odd shape on the downward slope. The manufacturer of the instrument was contacted for an explanation<sup>19</sup>. This is an artifact of the way the heat flow evolution is displayed. This feature is better understood when the heat flow is displayed as function of time. The KClO<sub>3</sub>/icing sugar mixture has a very sharp exothermic event at ~180°C. This event is so rapid that, if the sample size is large enough, the heat flow can overwhelm the system, causing the controller thermocouple to shut the heating system down. When the system recovers, the heater will start again, but there is some lag time, so the recorded temperature spikes and then decreases until the system catches up. When displaying the heat flow in the traditional manner with temperature on the x-axis, this appears to be a retrogression of the temperature. The exothermic event is so strong for KClO<sub>3</sub>/icing sugar; only very small sample sizes do not overload the system and do not show this retrogression.

The Ex<sub>2</sub> exothermic feature may be due to the oxidative decomposition of the icing sugar. Pure icing sugar, when examined in the DSC (not shown), exhibits a very sharp endothermic event at ~190 °C, assigned to the

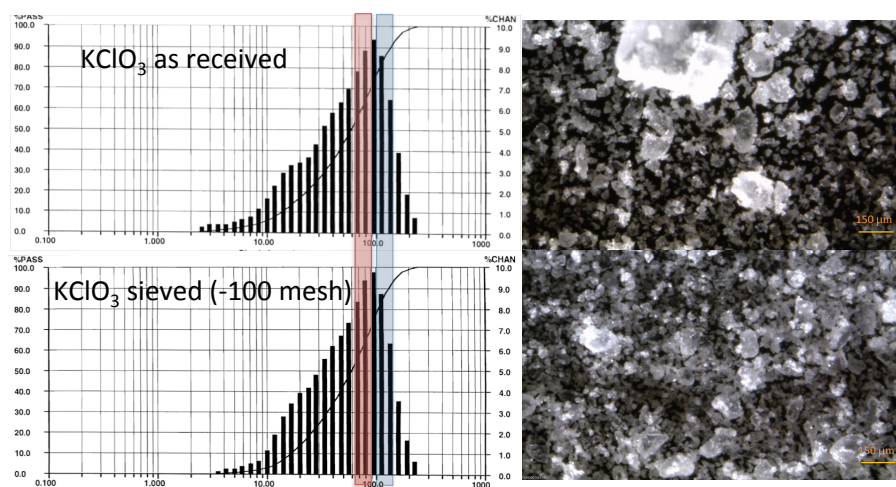


melting of the sugar; a broad endothermic event at  $\sim 220^\circ\text{C}$ , assigned to the decomposition of the sugar structure; and a very broad, weak exothermic event at  $\sim 340^\circ\text{C}$ , assigned to the oxidation of the residual char from the decomposition. In the pure sugar case, the DSC system has little or no available oxygen for an oxidative reaction (which would cause an exothermic event), except at higher temperatures when the sugar starts to lose oxygen. It is speculated that with the case where the oxidizer  $\text{KClO}_3$  is present, some of the icing sugar that is not consumed to give  $\text{Ex}_1$ , will start to decompose at the sugar decomposition temperature, but the by-products will be oxidized causing it to be an exothermic event, instead of an endothermic event.

Regardless of the mechanism, these results indicate that the low-temperature exothermic event is more appropriate for characterizing bulk samples of  $\text{KClO}_3$ /icing sugar mixtures since bulk samples will always be relatively homogeneous mixtures and not exhibit isolated pockets of materials. This indicates that KC-sugar is then as thermally stable as RDX and perhaps more energetic upon decomposition.

#### 4.4 Comparison of $\text{KClO}_3$ /icing sugar (AR) mixture with $\text{KClO}_3$ /icing sugar (-100) mixture

Table 10 also has SSST testing data on the previously reported  $\text{KClO}_3$ /icing sugar (-100) mixture<sup>2</sup>. This material was prepared from the same component materials shown in the Experimental section, but with the only difference that the  $\text{KClO}_3$  was screened through a 100-mesh sieve. The reasoning behind the comparison was to see if particle size effects of the oxidizer component will make any difference in SSST testing behavior. In retrospect, the size difference was not significant. A previous study<sup>18</sup> showed that as-received  $\text{KClO}_3$  can have very large particles due to agglomeration. Figure 2<sup>20</sup> shows this agglomeration has little impact on the particle size distribution of the materials in this study. Except for a few large particles visible in the photographs, the distributions look very similar. The agglomeration is very weak, so the solvent used in the size measurement breaks the large particles into the smaller material. Either these large particles are broken down readily, are selectively not tested because of size, or are very low in concentration to have little effect on testing the results, as shown in Table 10, and discussed below.



**Figure 2. Size distribution (left side) by laser light scattering and photograph (right side) of  $\text{KClO}_3$  as received and screened through a 100-mesh sieve. Also shown is the UAM specifications for 120-grit and 180-grit sandpapers.**

For impact sensitivity, LLNL, LANL, and IHD report about the same behavior from the two mixtures, although IHD reports the as-received mixture to be slightly more sensitive. AFRL did not test the 100-mesh sieved mixture.



For BAM friction, LLNL data indicates the as-received mixture more stable than the sieved mixture. An evaluation of stability using these values would indicate the as-received mixture is more stable than PETN, while the 100-mesh sieved material is less stable. LANL data indicates the as-received mixture is slightly less stable than the 100-mesh sieved material, but still finds it more sensitive than PETN. IHD data has comparable values between the as-received and 100-mesh sieved mixtures.

Only IHD has performed ABL friction testing of both mixtures. In both the TIL and  $F_{50}$  evaluations, the  $KClO_3$ /icing sugar (AR) mixture is significantly more stable than the  $KClO_3$ /icing sugar (-100) mixture, paralleling the results for BAM friction from LLNL.

For the ESD all the participants find the sensitivity to be comparable between the two mixtures, with LLNL showing that the mixtures have no sensitivity.

## 5 CONCLUSIONS

$KClO_3$ /icing sugar (AR) mixture was found through SSST testing to be a moderately sensitive mixture toward impact, friction, spark and thermal handling conditions—generally more sensitive than RDX, and on the order of sensitivity of PETN.

The proficiency study shows that the current equipment configurations and experimental methods used for RDX and  $KClO_3$ /icing sugar (AR) mixture, the impact results from LLNL tend to show materials to have more stability than the results from LANL, IHD, or AFRL. For friction and ESD, results from LLNL tend to show the materials to have more stability than the corresponding results from LANL, IHD, and AFRL. For thermal results, unlike in the case for RDX, where all the participants had results that were virtually identical, sampling issues are causing inconsistent results, similar to the results for the  $KClO_3$ /icing sugar (-100) mixture.

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## ACRONYMS AND INITIALISMS

-100	solids sized through a 100-mesh sieve
$\Delta H$	Change in enthalpy
ABL	Allegany Ballistics Laboratory
AFRL	Air Force Research Laboratory, RXQF
AN	ammonium nitrate
BAM	German Bundesanstalt für Materialprüfung Friction Apparatus
C	Carbon
$C_{12}H_{22}O_4$	Sugar
CAS#	chemical abstract services log number
$CO_2$	Carbon Dioxide
$DH_{50}$	The height the weight is dropped in Drop Hammer that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
DHS	Department of Homeland Security
DSC	Differential Scanning Calorimetry
DTA	Differential thermal analysis
ESD	Electrostatic discharge
$F_{50}$	For BAM Friction, $F_{50}$ , in kg, is by a modified Bruceton method, load for 50% probability of reaction; For ABL Friction, $F_{50}$ in psig/fps, is by modified Bruceton Method, load for 50% probability of reaction

fps	Feet per second
g	grams
H	Hydrogen
H <sub>2</sub> O	Water
HME	homemade explosives or improvised explosives
HMX	cyclotetramethylene-tetranitramine
HP/F	hydrogen peroxide/fuel
IDCA	Integrated Data Collection Analysis
IHD	Indian Head Division, Navel Surface Warfare Center
j	joules
KCl	Potassium chloride
KClO <sub>3</sub>	Potassium Chlorate
KClO <sub>4</sub>	Potassium perchlorate
LANL	Los Alamos National Laboratory
LLNL	Lawrence Livermore National Laboratory
MEKP	methyl ethyl ketone peroxide
N	nitrogen
O	oxygen
PETN	Pentaerythritol tetranitrate
psig	pounds per square inch, gauge reading
RDX	Research Department Explosive, 1,3,5-Trinitroperhydro-1,3,5-triazine
s	Standard Deviation
S/C	Silicon carbide
SNL	Sandia National Laboratories
SO/F	solid oxidizer/fuel
SSST	small-scale safety and thermal
TGA	Thermogravimetric Analysis
TIL	Threshold Initiation Level (TIL) is the load (in kg for BAM friction, in psig/fps for ABL friction, in joules for ESD) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level;
T <sub>max</sub>	Temperature maximum of peak
UN	urea nitrate

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